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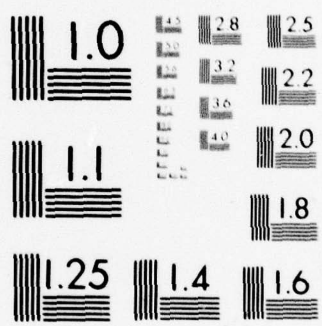


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# FRS COMPOSITES FOR ADVANCED GAS TURBINE ENGINES COMPONENTS

TRW
MATERIALS TECHNOLOGY





FINAL REPORT

Prepared Under Contract N62269-77-C-0217

Approved for public release; distribution unlimited

NAVAL AIR DEVELOPMENT CENTER
WARMINSTER, PENNSYLVANIA 18974
FOR
NAVAL AIR SYSTEMS COMMAND
DEPARTMENT OF THE NAVY
WASHINGTON, D.C. 20361



MAY 1979

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

A number of FRS design concepts involve use of varying volume fraction of fiber varying fiber diameter, and cross-ply construction. Key properties of such composites were evaluated to determine whether there are any adverse effects. Based upon elevated temperature tensile, creep, and thermal fatigue testing, equivalent mechanical properties are obtained if placement of plies is varied in a symmetrical layup.

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Abstract (contd)

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Techniques for fabrication of fully dense MA 956E-matrix composites without fiber damage were demonstrated.




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# FOR
# ADVANCED GAS TURBINE ENGINE COMPONENTS

By

D. M. Essock

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Prepared Under Contract N62269-77-C-0217

Naval Air Development Center
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For

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## FOREWORD

This final report describes the work performed for the Department of the Navy under NADC Contract N62269-77-C-0217. It is published for information only and does not necessarily represent the recommendations, conclusions or approval of the Navy.

This contract with TRW Inc., 23555 Euclid Avenue, Cleveland, Ohio 44117 was performed with Mr. Irvin Machlin, AIR-52031B, Naval Air Systems Command, Washington, D. C. 20361 as technical consultant.

At TRW, Ms. D. M. Essock was the program manager reporting to Mr. J. N. Fleck, Manager of the Metal Composites and Powder Technology Section. Other TRW personnel contributing to the program were Mr. L. E. Chojnowski, panel fabrication and testing, Mr. J. W. Sweeney, thermal fatigue testing, Messrs. C. A. Harris and C. A. Tyndall, mechanical testing.

Prepared by D. M. Essock

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Approved by J. N. Fleck

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and

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Materials Development
Department

## ABSTRACT

A number of FRS design concepts involve use of varying volume fraction of fiber, varying fiber diameter, and cross-ply construction. Key properties of such composites were evaluated to determine whether there are any adverse effects. Based upon elevated temperature tensile, creep, and thermal fatigue testing, equivalent mechanical properties are obtained if placement of plies is varied in a symmetrical layup.

Based upon limited data, a $\pm 15$ degree laminate is superior to a 0° laminate in shear but inferior in creep and thermal fatigue resistance. Longitudinal tensile strength is comparable to, or slightly less than, that for a unidirectional composite.

Techniques for fabrication of fully dense MA 956E-matrix composites without fiber damage were demonstrated.

## 1.0 INTRODUCTION

In previous work, a refractory wire reinforced FeCrAlY system having a desirable combination of properties for advanced gas turbine engine applications was developed. These include oxidation/corrosion resistance, creep strength, stress rupture, thermal fatigue resistance, and additionally, a lower cost potential than other advanced candidate systems.

The W-1$ThO_2$/FeCrAlY system developed under NASC, NASA and TRW internal funding has been selected for a 1st generation FRS prototype blade design and fabrication study. This first generation system has potential for operating at up to 1148°C (2100°F) metal temperatures and exhibits up to a threefold specific strength advantage (1000-hour creep rupture) over the best competing metallic systems, oxidation/corrosion resistance such that protective coatings are not required, and adequate resistance to thermal cycling based on 1000-cycle 27-1204°C (80°-2200°F) tests. Preliminary cost estimates have also indicated that components such as FRS turbine blades could be fabricated at lower costs than other competing systems, such as directionally solidified eutectics(1).

Important areas for investigation relating to turbine component design requirements, which are not covered in other programs, include physical and mechanical property data evaluations on mixed filament diameters, improved shear strength matrices, "bi-metal" constructions and variable fiber contents. With additional property data, it should be possible to factor in other diffusion controlled, time dependent effects, such as growth or reaction zones, etc. Advanced systems with higher temperature capabilities, incorporating coated fibers or potentially low cost non-metallic fibers, should also be considered for future generation FRS systems.

This current program was designed to investigate some of these advanced concepts relating to FRS composite design and component fabrication.

## 2.0 BACKGROUND

The continuing objective of this program has been to develop an oxidation/corrosion resistant metal-matrix fiber reinforced composite system designed for advanced gas turbine engine components which are capable of operating, without a protective coating, at temperatures up to 1200°C (2400°F).

A FeCrAlY alloy was selected for the matrix alloy based on known properties which include: excellent oxidation/corrosion resistance up to 1370°C (2500°F), good ductility, high melting point, low density, good fabricability and low cost.

In the initial program(2), fabrication parameters were developed and screening studies performed using a variety of potential reinforcements, including SiC, $Al_2O_3$, W and Mo, and reaction barrier coatings. Based on elevated temperature compatibility, preliminary stress rupture data and cost and availability, refractory metal wires (tungsten alloy, molybdenum alloy) were identified as having greatest potential as reinforcements for the first generation FRS systems. A W-1$ThO_2$/FeCrAlY composite system was subsequently shown to have potential long term (>1000-hour stress rupture) life at temperatures up to 1150°C (2100°F). Extended temperature capability would require the use of reaction barrier coatings and thermodynamically stable carbides TiC, TaC, HfC were identified as promising diffusion barriers coatings.

Subsequent work(3,4) showed that the first generation system had moderately good thermal fatigue resistance, very high density-compensated creep rupture strengths (2 1/2 times those of D.S. eutectics at 1040°-1150°C), excellent oxidation resistance typical of the FeCrAlY matrix alloy and adequate strengths (angle-plied construction) at 650°-760°C to withstand typical blade root stresses. Cost estimates for turbine blade manufacturing processes(1) have indicated a very favorable position for W/FeCrAlY components compared to even conventional D. S. superalloys.

In the most recent work(5), data on low cycle fatigue behavior and impact properties were developed and the feasibility of hollow airfoil fabrication demonstrated using a preconsolidated monotape and leachable core technique.

## 3.0 PROGRAM PLAN

### 3.1 Effect of Improved Shear Strength Matrices

Centrifugal stresses developed in a DSE or FRS turbine blade will be transmitted to fibers in the root area by a shear load transfer mechanism. For a single fiber pullout specimen, it was shown[6] that the critical aspect ratio $\frac{L_c}{D}$ is related to the fiber fracture stress and matrix shear stress by

$$\left(\frac{L_c}{D}\right)_t = \left(\frac{\sigma}{4\tau}\right)_t$$

Where $L_c$ = critical fiber length

$D$ = fiber diameter

$\sigma$ = tensile stress for fiber failure in time t

$\tau$ = matrix shear stress for failure in time t

Thus, it is seen that there is a 4:1 relationship between matrix shear strength and critical fiber length for pullout. It should be observed that the above relationship is regarded as conservative for multi-fiber composites where tri-axial restraints on the matrix may exist. Cross plying results in additional restraints to fiber pullout. It is felt that at least modest improvements in matrix shear strength could result from the use of oxide dispersion strengthened material and have significant effects on blade root design.

### 3.2 Effect of Varying Volume Fraction of Filament

Because of the high density of the tungsten reinforcement, the most efficient design for an FRS blade will probably involve a concept such as that shown in Figure 1. An objective of this study is to investigate the effects of varying volume fraction on key properties.

### 3.3 Effect of Mixed Filament Diameters

One advanced FRS concept involves the use of smaller diameter (>0.015") filaments in airfoil locations where higher stresses and lower temperatures prevail. Higher volume fraction loadings would be possible with smaller diameter fibers (0.004-0.010") since smaller ply thicknesses would allow balanced angle-ply buildup in relatively thin areas in a hollow blade. It will be necessary to evaluate the creep and fracture behavior of these composites with mixed diameter fibers. Tensile and creep tests will be performed on composites containing fibers of different diameters.

### 3.4 Advanced Reinforcement Materials

The W-1$ThO_2$/FeCrAlY system has been selected as the first generation FRS material. The potential, in terms of specific strength (i.e., density normalized strength) for FRS materials based upon lower density fibers such as SiC is

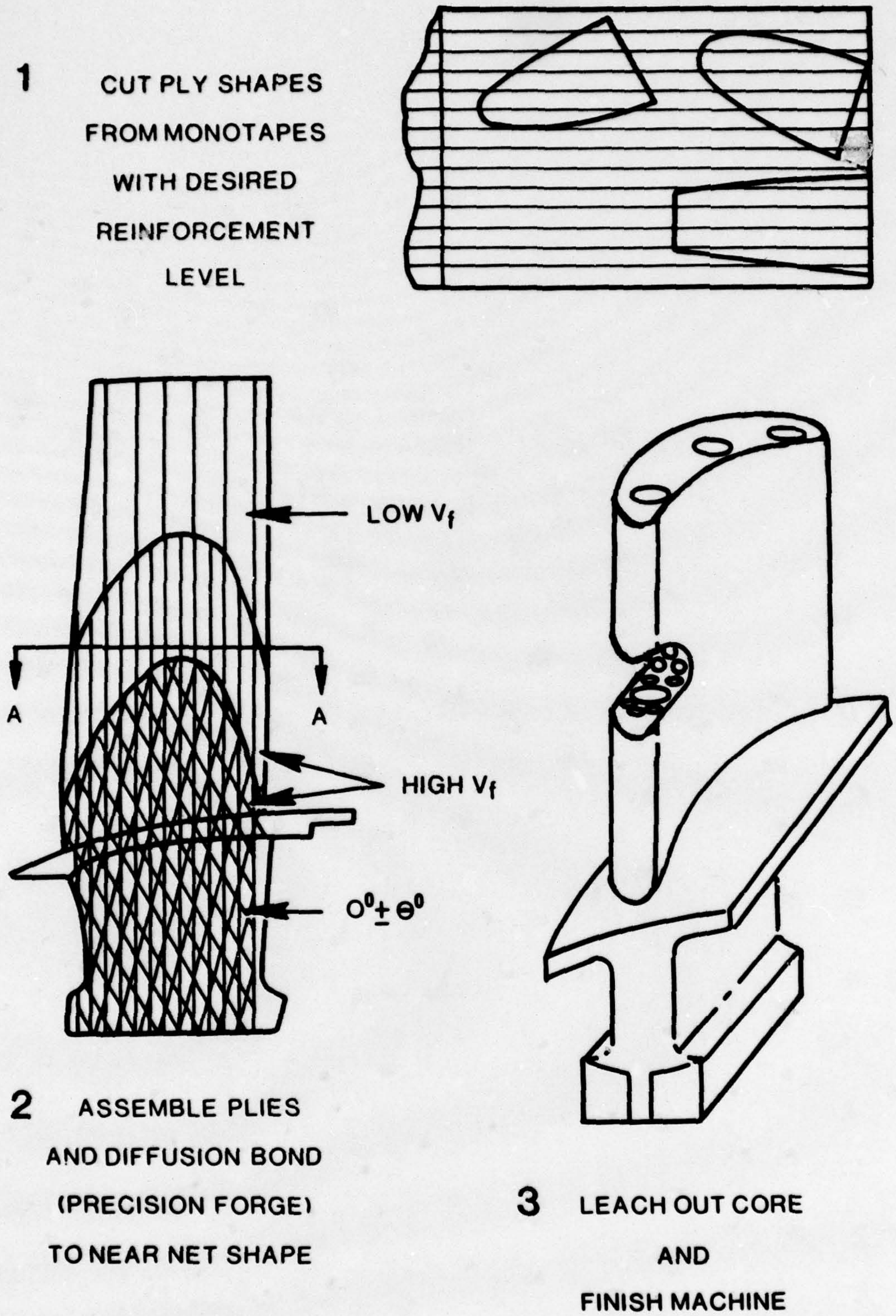



Figure 1. Schematic Illustration of Stages in the Fabrication of FRS Blades with Internal Cavities and Variable Fiber Reinforcement Level.

too great to ignore. In previous work, it was clearly demonstrated that, to be useful in FRS, SiC must be coated to prevent fiber-matrix reaction[2]. The state-of-the-art in fiber coating is advancing in both quality and variety of coatings. A minor objective of this effort was to assess the applicability of coated SiC in FeCrAlY.

### 3.5 W-1$ThO_2$/FeCrAlY Characterization

Critical property tests will be performed to further characterize this first generation FRS system. This will include shear, elevated temperature tensile, thermal and low-cycle fatigue and effect of thermal cycling on residual tensile strength.

## 4.0 PROCEDURES AND RESULTS

### 4.1 Material Procurement

#### Matrix Alloy

The nominal composition of the selected matrix alloy is Fe-20-24Cr-5Al-1.0Y (elements in weight percent). This was procured from Special Metals, Udimet Powder Division of Allegheny Ludlum in the form of pre-alloyed 2541 powder made by the argon atomization process. The majority of the powder being used on the program is screened into the particle size range -80 + 500 mesh.

The 2541 alloy is characterized as having a ferritic matrix with dispersed $YFe_9$ particles and alpha-prime (Fe-Cr) precipitates. The alpha prime grows (overages) at elevated temperature, and therefore, strength drops. If an inert dispersoid is added, theoretically the strength could be retained to higher temperatures. This is the basis for International Nickel's MA 956E (Fe-20Cr-4.5Al-0.5Ti-0.5$Y_2O_3$). The oxide becomes the strengthening phase.

It is significant to note that oxidation resistance of FeCrAlY is not affected by additions of 1 to 4 volume percent oxide[7].

Sheets of MA 956E of two thicknesses, viz 0.025 and 0.015 inch, were obtained from International Nickel. However, since thinner material is needed for a high volume loading in a composite, some of the 0.025 inch material was further rolled at TRW. By use of a series of light reductions, we were able to successfully reduce 0.025 sheet to 0.008 inch and maintain flatness and surface integrity.

#### Reinforcement

The selected fiber reinforcement to be used for this program is W-1$ThO_2$ of 0.015, 0.010 and 0.005-inch diameter. This is commercially available material and may be procured from at least two vendor sources. For the current program, this material was procured from GTE Sylvania to standard lamp filament specifications in terms of surface condition and straightness.

#### Advanced Fiber Reinforcements

One of the continuing tasks in this work has been the evaluation, at least on an exploratory basis, of advanced reinforcement systems which may represent higher temeprature capabilities, higher specific strength, or low cost potential.

A number of reaction-barrier-coated fibers were made available by Dr. Iqbal Ahmed of Watervliet Arsenal, and filament/matrix interaction studies were conducted on several of these.

## 4.2 Test Specimen Panel Fabrication

Fabrication procedures have been discussed in detail in previous reports (2, 3, 4, 5). The process consists of fiber collimation by drum winding, converting the pre-alloyed powder to sheets of specified thickness and density by use of a fugitive plasticizer, and consolidation of assembled layers of fibers and matrix cloth by inert atmosphere diffusion bonding. For these materials, typical hot pressing parameters are 1000°-1120°C (1850°-2050°F) at 140-207 MPa (20-30 ksi) for 30-90 minutes. Molybdenum alloy dies were used with induction heating.

Good fiber distributions with a fairly uniform hexagonal array and no fiber/fiber contacts were obtained by this process. Other advantages of this process are: 1) almost any desired matrix composition may be selected and 2) no degradation of fiber properties occurs since the fabrication temperatures are no higher than projected use temperatures.

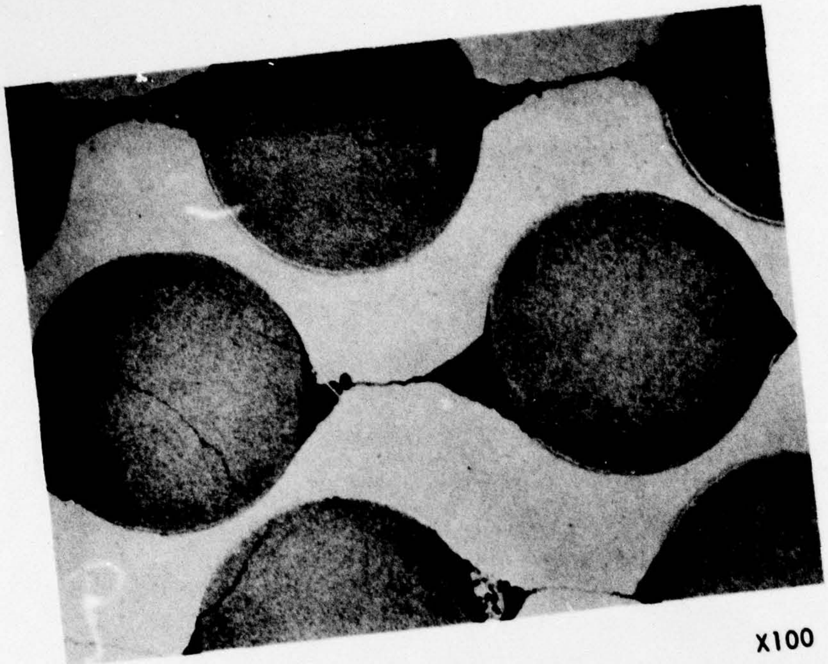
## 4.3 ODS Matrix Alloy Investigation

A sample of 0.025-inch thick MA 956E was obtained from International Nickel.

For an initial evaluation, this was reduced to nominally 0.0075 inch by chem-milling. A four ply panel was assembled with mats of 0.015 inch $W\text{-}1ThO_2$ wire. This was pressed in a "normal" cycle, i.e., under a clamping load up to 1800F, then 25 ksi applied as temperature was increased to 2025F, and temperature and pressure held for 45 minutes. Consolidation of the matrix was poor, and there was extensive fiber damage, as can be seen in Figure 2. Obviously, the flow stress of this material is sufficiently high that this bonding cycle is no longer applicable. A repeat of this trial yielded the same results.

Microhardness measurements taken on the MA 956E before and after pressing indicate a substantial drop in hardness occurred as a result of the processing cycle used (as-received hardness, 450.4 KHN; hardness after processing, 371.1 KHN). The as-polished microstructure shows essentially no change (see Figure 3), but, in the etched condition, structural coarsening is apparent. This can be seen in Figure 4.

Another 3-ply panel was prepared in the same manner, but the pressing cycle was modified. Specifically, the clamp load was maintained to the 2050F bonding temperature to further soften the matrix prior to application of full load to preclude fiber damage. Also, time at temperature and pressure was increased from 45 to 90 minutes. Most of this panel was fully consolidated, however, there were some residual voids and unbonded areas (for example, see Figure 5-A). In the well-consolidated areas, fiber cracking persisted (see Figure 5-B) albeit less severe than in the previous panel.



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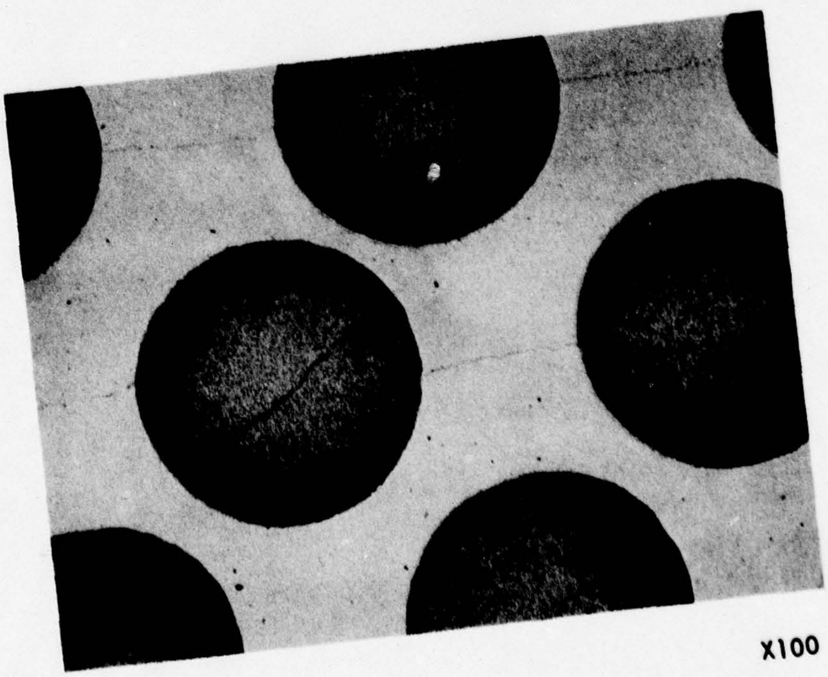

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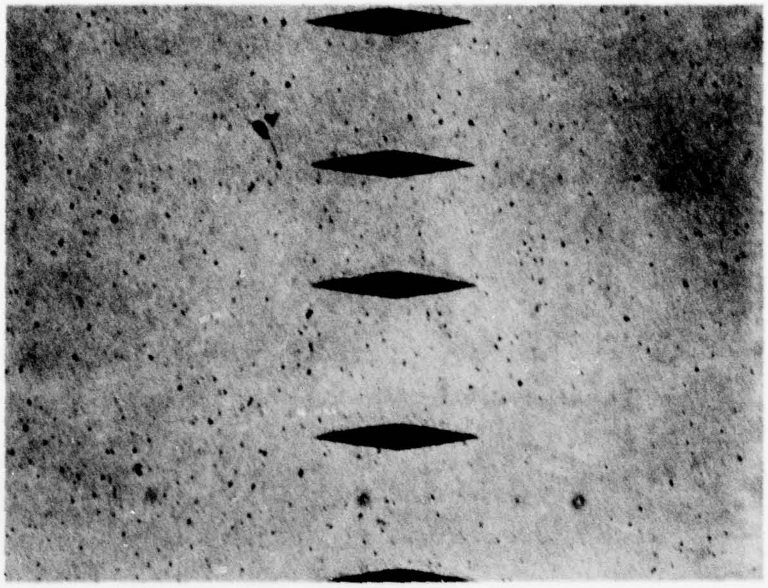
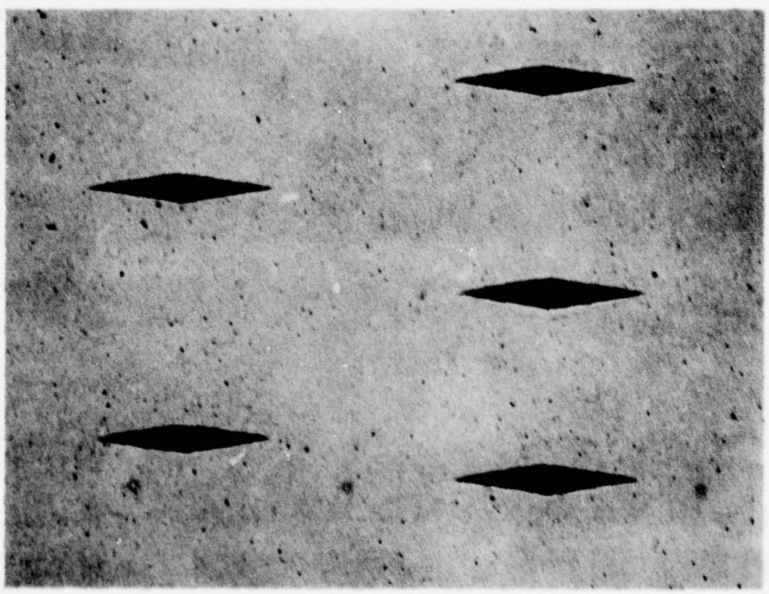
Figure 2. Initial Trials on W/MA 956E Composite. The top photomicrograph shows lack of consolidation and the bottom poor matrix-matrix bonding; both show fiber damage.

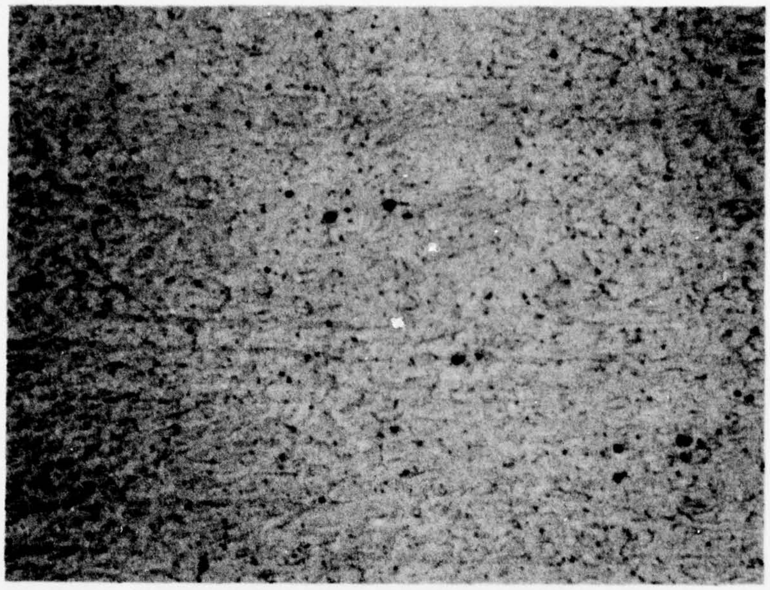


A. Before Pressing

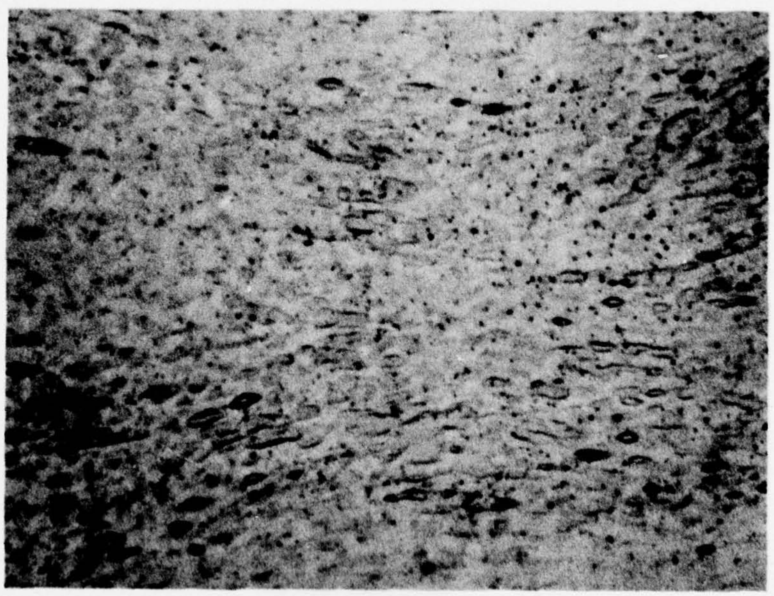


B. After Pressing

Figure 3. MA 956E Microstructure, As Polished (400X)

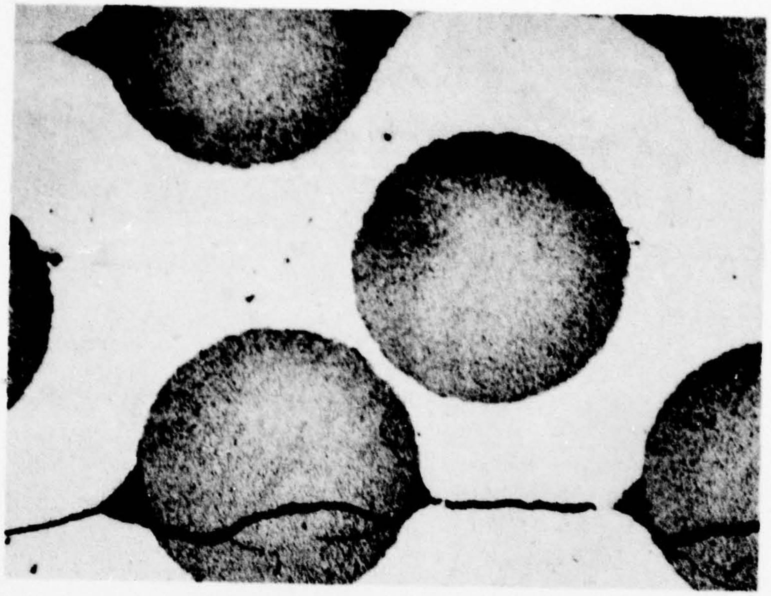


A. Before Pressing

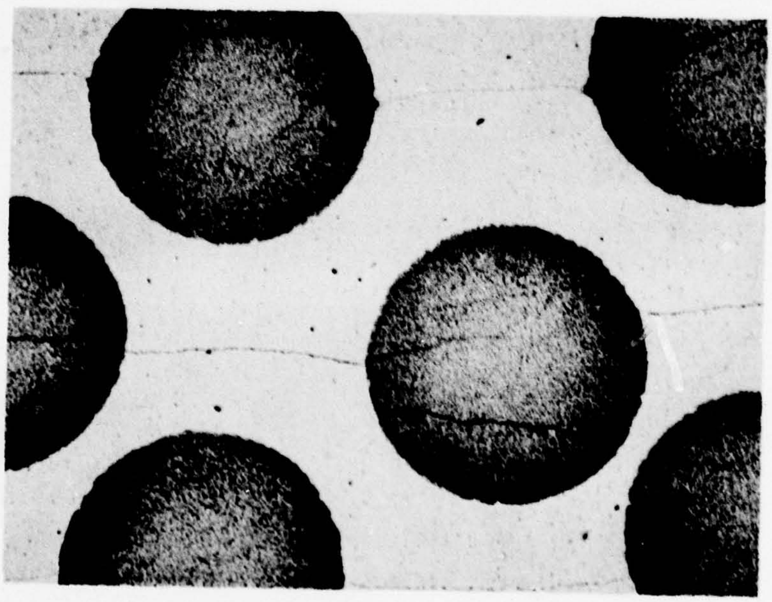


B. After Pressing

Figure 4. MA 956E Microstructure, Etched (800X)



A. 100X



B. 100X

Figure 5. W/MA 956E Composite Pressed at Higher Temperature.

To further reduce fiber damage, a FeCrAlY powder layer was introduced between the MA 956E foils. For this trial, the fibers were 0.004 inch and the MA 956E was reduced to 0.005 inch. The FeCrAlY was in the form of a 0.002 inch thick transfer tape produced commercially by Vitta Corporation using a procedure developed for braze-alloy transfer tapes. The powder density is less than 50% (lower than our standard powder cloth) and the binder content rather high, but, in a vacuum bake-out experiment, it was extracted leaving no residue.

The bonding cycle was the same as that for the previous panel. As can be seen in Figure 6, this resulted in a well consolidated panel free from fiber cracking, as can be seen in Figure 6-A. A very close examination at a higher magnification (Figure 6-B) reveals the FeCrAlY layer between the MA 956E foils. In the photograph, this interface is horizontal and in line with the fiber center line.

This was the final panel prepared in this series, and tensile shear tests were planned. Unfortunately, the panel was destroyed during specimen machining, and no data were obtained.

### 4.4 Effect of Varying Volume Fraction

A number of conceptual selective reinforcement concepts are based upon the use of a variable volume fraction of wire to minimize weight and cost. This task was intended to assess whether this resulted in any unusual behavior. Panels were prepared from monotapes, as this approach is most likely to be used for actual hardware.

A series of monotapes containing 20, 30, and 50v/o of 0.008 inch $W\text{-}1ThO_2$ wire were prepared. From these, two sets of panels were prepared. One set contained the following v/o plies: 50, 30, 20, 20, 30, 50; the other 20, 30, 50, 50, 30, 20.

Specimens were machined into the standard elevated temperature test geometry, shown in Figure 7. Tensile tests were performed at 2000F at a constant cross-head speed of 0.020 inch/minute. Results were as follows:

| Type of Construction | UTS | Approximate Elongation* |
|---|---|---|
| A. High v/o on outside | 48,000 psi | 5.8% |
| B. High v/o on inside | 47,100 psi | 5.2% |

From this, we conclude that, for a symmetric layup, it makes no difference whether the high volume fraction plies are on the surface or mid plane.

Creep tests were also run at 30 ksi and 2000F. These results are shown in Figure 8. Until a temperature control problem due to a malfunctioning heater element forced one run to be aborted, the two layups appear to exhibit the same creep behavior.

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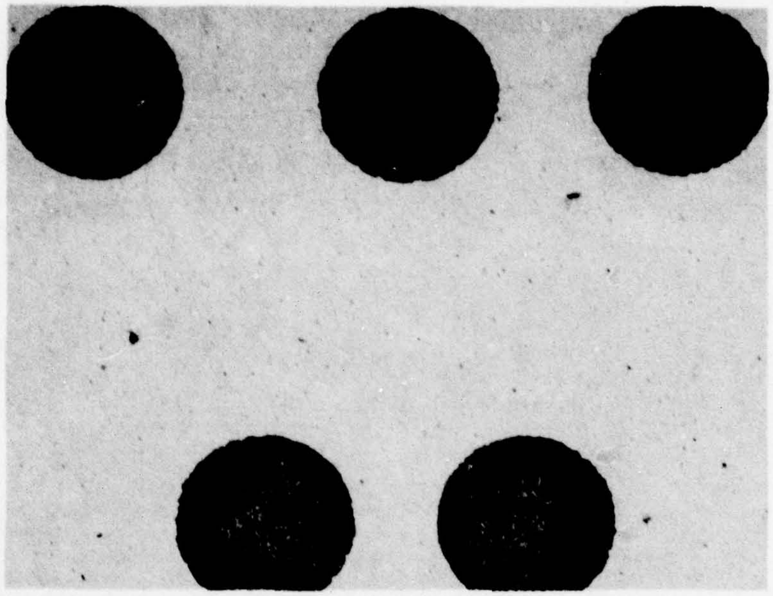
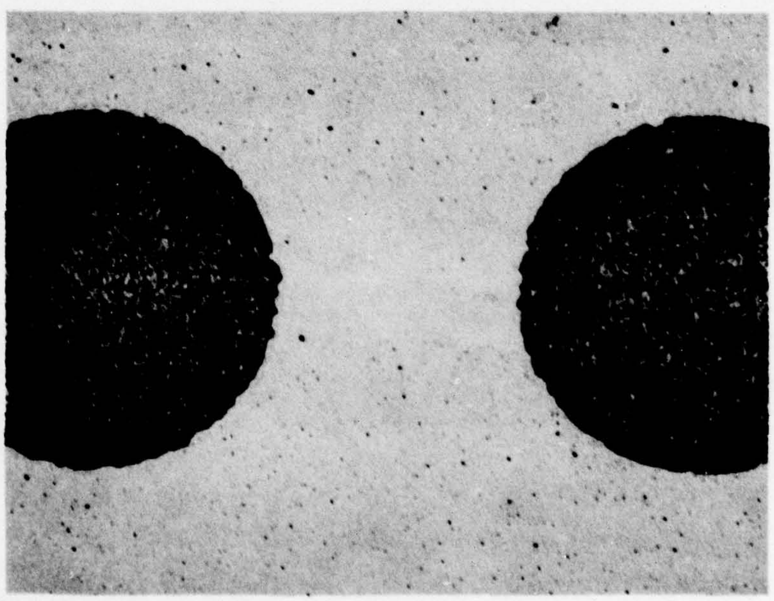
* Measured by fitting broken specimen halves back together.

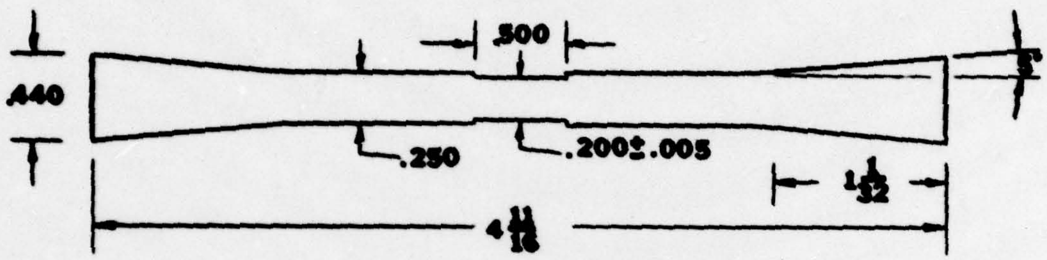


A. 250X

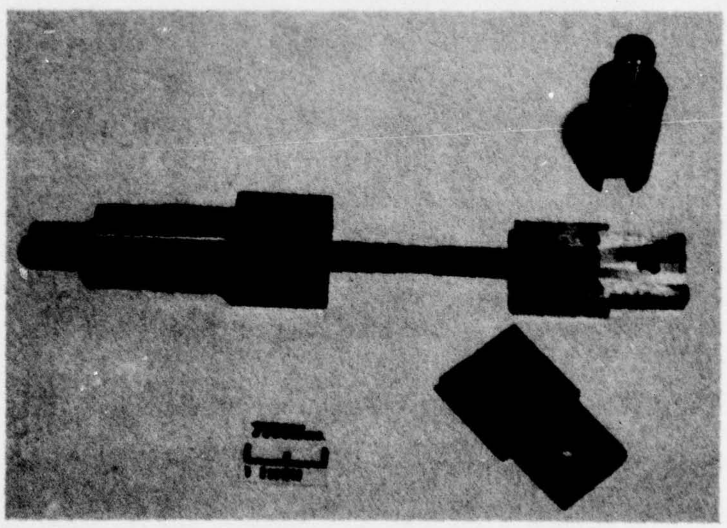


B. 500X

Figure 6. W/MA956E Composite Prepared with Powder Layer Between Foils.




A. Specimen Design



B. Grip Assembly and Machined Specimen

Figure 7. Standard FRS Test Specimen.

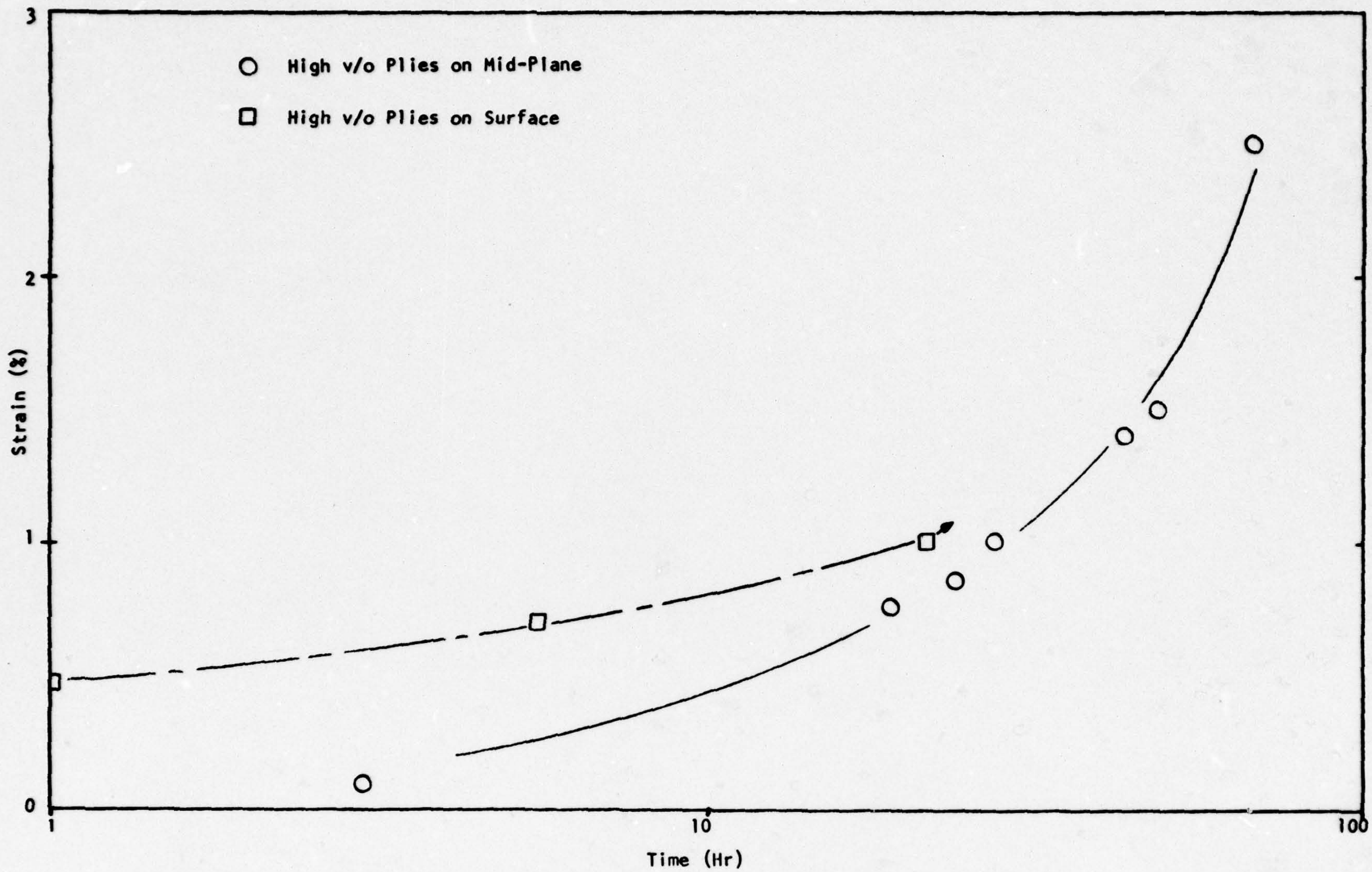



Figure 8. Creep of Varying v/o Laminates.

## 4.5 Effect of Mixed Filament Diameter

The choice of a fiber diameter for FRS involves a number of factors. In terms of cost, larger diameters are least expensive. However, smaller diameters are stronger for comparable processing schedules. Smaller fiber diameters mean thinner monotapes, hence more plies to achieve a given thickness. This translates into higher cost. Some airfoils, or airfoil sections, are so thin that only very small diameter fibers can be accommodated and still preserve a minimum thickness of FeCrAlY on all sides of the fibers. Other large components could readily be prepared from rather large diameter fibers.

Under a current NASA Contract (NAS 3-20390), the effect of fiber diameter on key properties is being established. The objective of this task was to determine whether mixed fiber diameters within a given panel posed any problems.

The monotape approach was also used for these trials. Nominally 50v/o monotapes were prepared with 0.004, 0.008, and 0.010-inch diameter W-1$ThO_2$ fiber. One series was prepared with the following fiber diameter stacking sequence (in mils): 10, 8, 8, 4, 4, 8, 8, 10; the other was: 4, 8, 8, 10, 10, 8, 8, 4.

Specimens were machined into the geometry shown earlier in Figure 7 and tensile tested at 2000F. Results are noted below.

| Type of Construction | UTS for Nominal 50v/o | Approx. Elongation |
|---|---|---|
| C. Large fiber dia. on outside | 86,900 psi | 7.8% |
| D. Large fiber dia. on inside | 73,500 psi | 6.8% |

This appears to be a significant difference. However, the panels were not identical in thickness. The Type C panel was 0.0585 in. thick whereas the Type D was 0.063 in. thick. This means the volume fractions were not identical. Average volume fractions for the two types of panels, as determined by fiber count, were 62% and 53% for the Types C and D, respectively. When normalized to a constant 62% volume fraction, the strength of the Type D panel becomes 86,000 psi. Thus, we conclude that, for a symmetric layup, the stacking sequence in a mixed diameter composite makes no difference, and mixing diameters has no adverse effect on properties.

Creep tests were also run on these materials at 30 ksi and 2000F. Results are shown in Figure 9. Again, there is no difference between the two laminate types.

These curves are decidedly different from those shown in Figure 8, and appear to show a cessation of creep after about 75 hours. As of this writing, the reason for the difference in creep behavior of the Types A and B versus Types C and D panels is unknown. The volume fractions of the two were quite different, however. The Types A and B average less than 30v/o, whereas the Types C and D average well over 50v/o. Both sets of creep tests were run at 30,000 psi, however. This same unusual behavior has also been observed on TFRS at 20 and 35v/o under a NASA program[8].

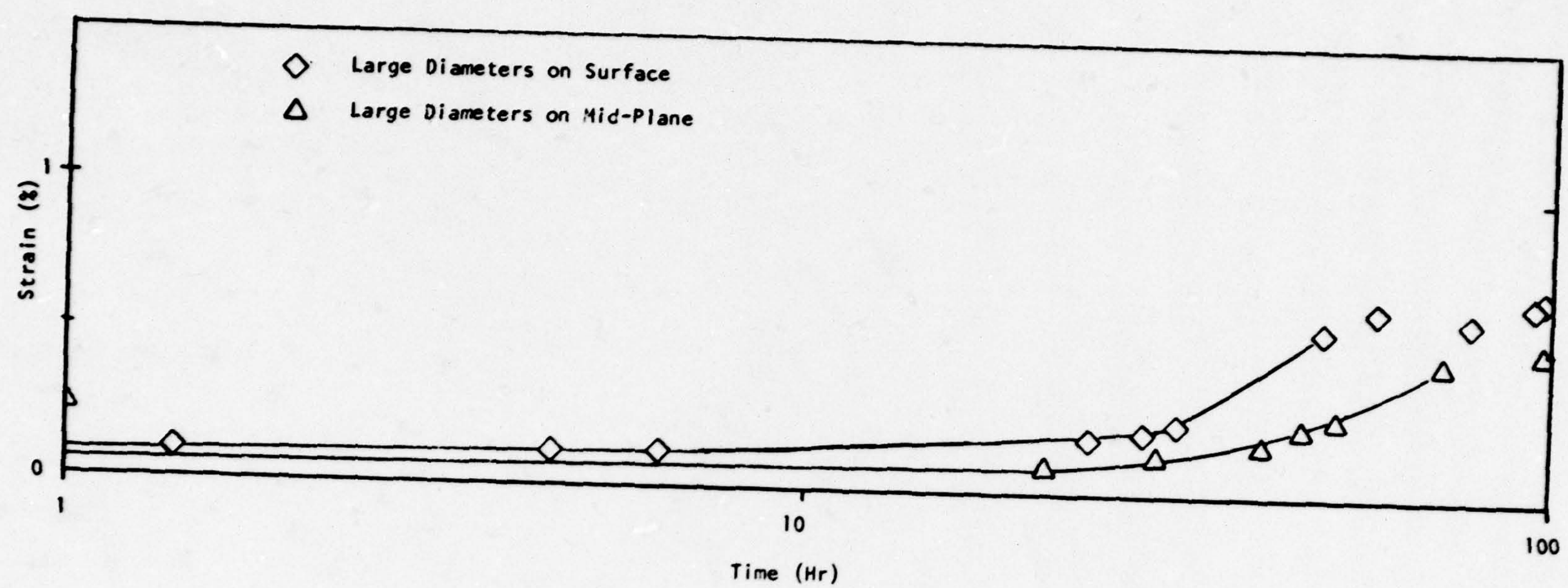



Figure 9. Creep of Varying Fiber Diameter Laminates

## 4.6 Effect of Angle-Plied Construction

Much of the characterization of FRS materials to date has been performed on 0° (unidirectionally reinforced) materials. The objective of this task was to evaluate the effect of angle-plied construction on some critical properties.

### 4.6.1 Elevated Temperature Tensile Strength

A five-ply, nominally 35v/o 0.010-inch W-1$ThO_2$ panel was prepared with the following stacking sequence: +15°, -15°, 0, -15°, +15°. The ultimate strength was 45,200 psi at 2000F and the approximate total elongation was 4.0 percent.

These strengths are comparable to those of unidirectional composites having comparable volume loading of fibers. For example, the Types C and D composites, normalized to 35v/o fiber, have strength on the order of 48,000 psi. This observation that longitudinal strength is degraded but little with low-angle cross-plying is consistent with the behavior of other metal matrix composites.

### 4.6.2 Creep Behavior

Creep testing was performed at 30,000 psi and 2000F on nominally 35v/o materials. Based upon a single data point, the ±15 degree laminate exhibits an accelerated creep, as compared with a unidirectional material (see Figure 10).

Similarly, the Larson-Miller parameter for the ±15° material is substantially lower than values previously reported for 0° FRS (see Figure 11). For comparison, values for the varying volume fraction (Types A and B) panels are also shown. The total volume fraction in these latter panels is lower than that covered by the reference data, and therefore, we conclude that the materials are of equivalent quality levels.

These results indicate a potential area of concern in designing with angle-plied FRS, and further work is needed in characterization and optimization creep/rupture of other than unidirectional laminates.

### 4.6.3 Shear Strength Vs Filament Orientation

As discussed earlier, high shear stresses will develop in the turbine blade root area and it is necessary to know the limiting stresses for fiber pull-out at temperatures representative of turbine blade root attachments.

Preliminary tests were carried out on W-1$ThO_2$/FeCrAlY specimens having different fiber distributions at 648° and 760°C (1200° and 1400°F). The double notch shear specimen was used; this provides in-plane or interlaminar strength data. Specimens having uni-axial (0°) and ±15° angle-ply constructions were tested. The data are listed in Table 1, and Figure 12 shows specimens after testing, illustrating the failure modes.

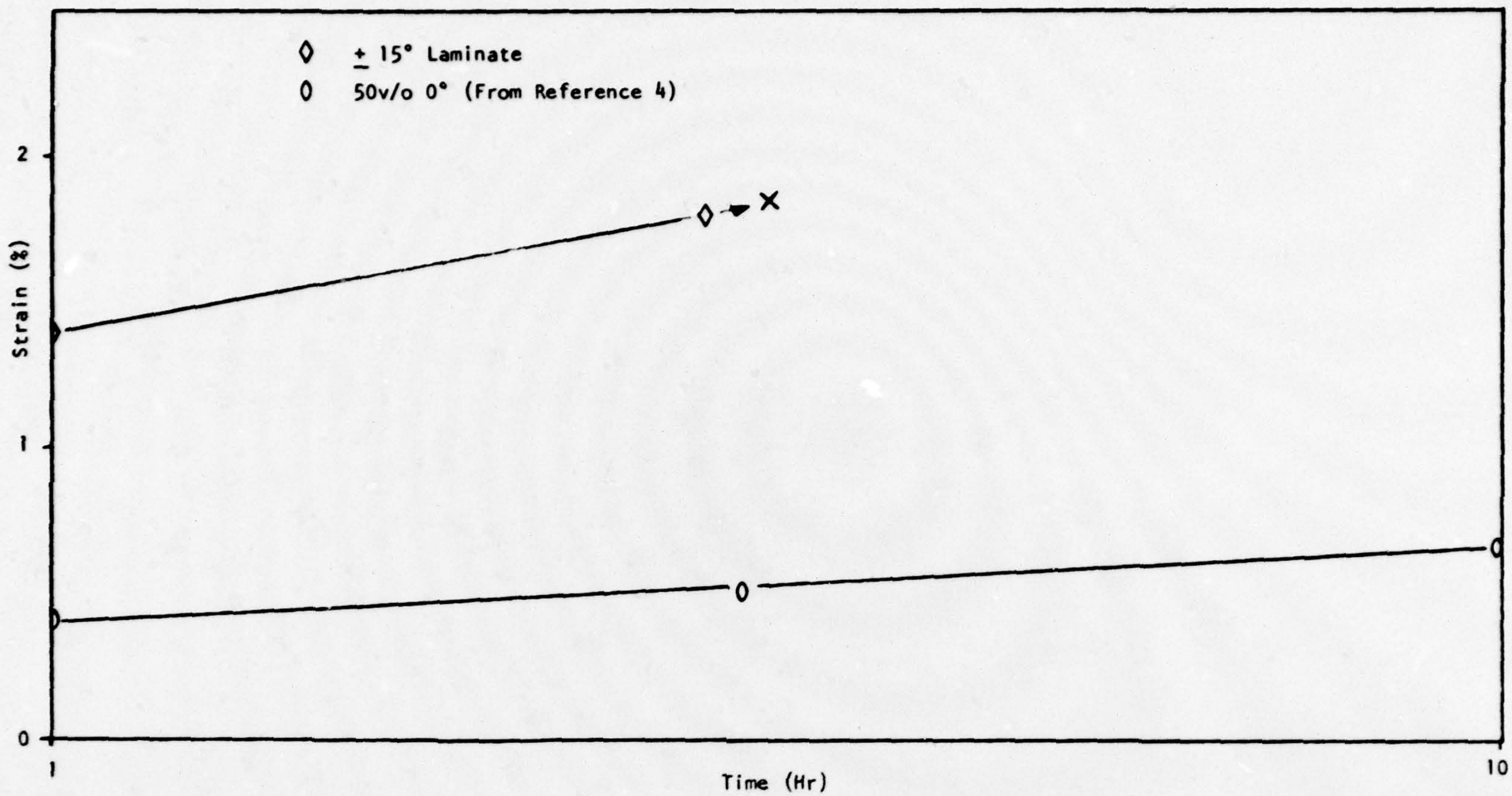



Figure 10. Creep of ±15° Laminate

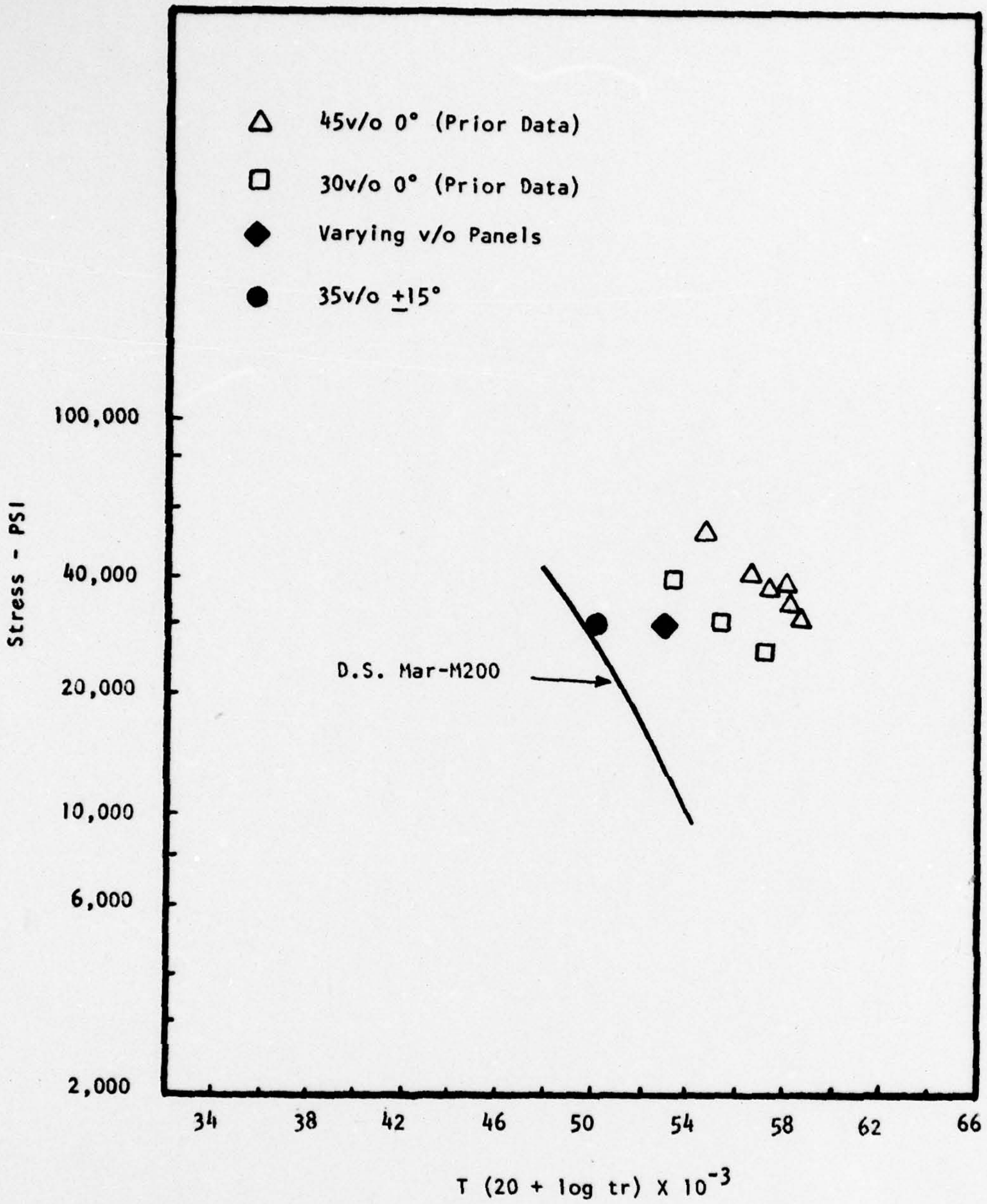
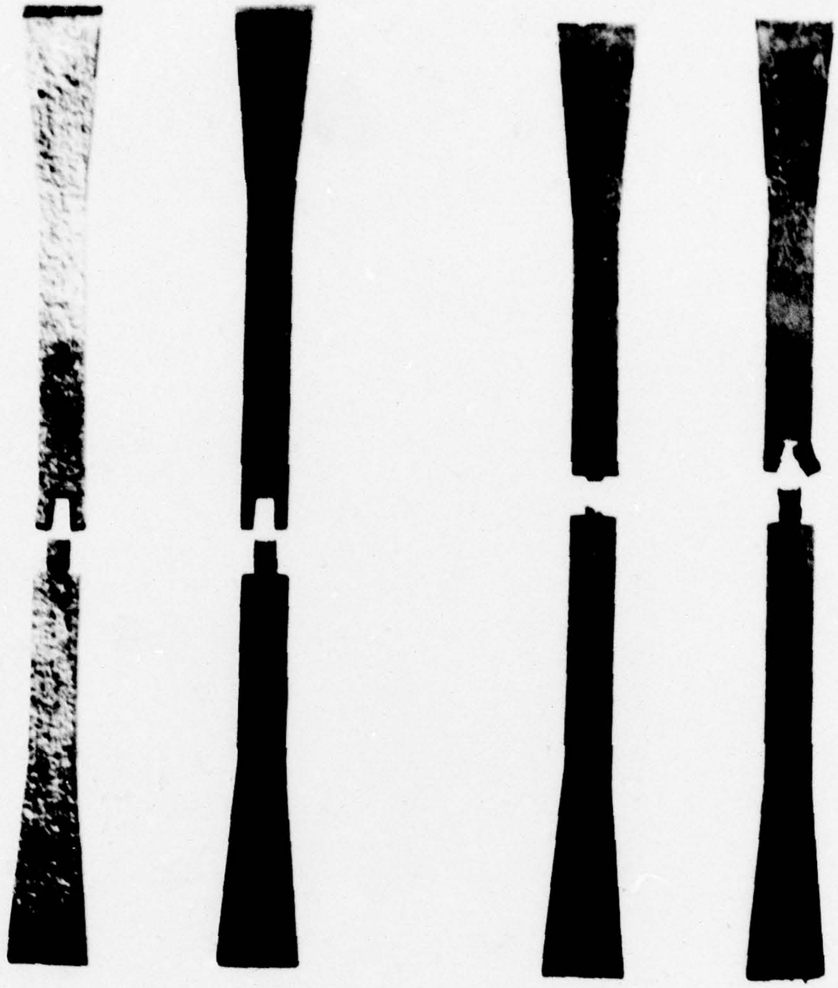



Figure 11. Larson-Miller Parameters for Varying Volume and Angle-Plied Panels.



648°C    760°C    648°C    760°C

Fiber Orientation 0°    Fiber Orientation $\pm 15°$

Figure 12. Fracture Mode in W-1ThO$_2$/FeCrAlY Double Notch Shear Specimen with Different Fiber Orientations.

## TABLE I

### Shear Strength Test Data on W-1%$ThO_2$/FeCrAlY

| Specimen I.D. | Fiber Orientation | Test Temperature °C | Test Temperature °F | Shear/Tensile Strength KSI | Failure Mode* |
|---|---|---|---|---|---|
| 2-9-0/1 | 0° | 648 | 1200 | 22.2 | S+T |
| 2-9-0/3 | " | " | " | 25.1 | S |
| 2-9-0/2 | " | 760 | 1400 | 14.4 | S |
| 2-7-15/1 | ±15° | 648 | 1200 | 78.4 | T |
| 2-7-15/2 | " | " | " | 80.75 | T |
| 2-7-15/3 | " | 760 | 1400 | 19.9 | S+T |
| 2-7-15/4 | " | 760 | " | 19.0 | S |

* S = Shear Failure

T = Tensile Failure

S+T = Mixed Failure

Specimens with uni-axial (0°) fiber orientation failed in shear at both temperatures whereas the specimens with ±15° reinforcement exhibited tensile failures at 648°C and mostly shear failures at 760°F. Some rotation towards the stress axis of stressed fibers closest to the center notch was necessary to allow failure by shear in the angle plied material, and there was clearly a temperature dependent matrix strength effect. It would also be expected that there would be a specimen size effect particularly with the angle plied material. The data in Table I indicate a 30% increase in tensile shear strength for ±15° reinforcements (for this specimen configuration) at 760°C while at the lower temperature where fiber rotation could not occur, tensile failures occurred.

## 4.7 Effect of Thermal Cycling W/FeCrAlY under Stress

In a previous program[5], W-1ThO$_2$/FeCrAlY specimens were subjected to thermal plus mechanical stress low-cycle fatigue testing. The results were reported in the Final Report. To complete the story, these same specimens have been tensile tested at room temperature to determine their residual strength.

Results are presented in Table II. For comparison, data for material thermally cycled under no load are also included. Note that the temperatures were lower in the combined stress tests. The net loss in strength was comparable to that experienced on thermal cycling at 2000F.

In this test, the ±15° laminates exhibited no unusual strength loss as compared with 0° materials.

Additionally samples were fabricated using variable $V_f$, fiber diameter, and ±15° orientation in the same scheme presented for the tensile and creep specimens. More stringent testing parameters were used for the low cycle and thermal fatigue tests. The fatigue tests were conducted in argon with a temperature cycle of 70-2000°F, and low cycle fatigue tests also used 0-50 ksi in-phase tensile stress cycles.

Table III showed that thermal fatigue results were excellent for varying fiber diameter specimens. After the 1000-cycle thermal-mechanical test, specimens had not cracked or delaminated. Varying volume fraction tests showed no differences between the large diameter fibers in the outer or inner plies, yet no conclusive test results were obtained due to temperature control problems. Only one sample of the ±15° angle ply specimen was tested and it had a comparatively shorter life. Equipment problems prevented any interpretations from being drawn from these low cycle fatigue data.

## 4.8 Test Procedures

Because of the low strength of FeCrAlY at elevated temperatures, testing of these materials poses some problems. For example, Figure 13 shows an exploded view of a grip/specimen assembly after an 1800F tensile test. An appreciable amount of deformation and slippage is evident. Nevertheless, a gage section failure was obtained.

TABLE II

Effect of Thermal Cycling on Residual Strength

| Orientation | Exposure | Room Temperature Tensile Strength % of Virgin Strength Remaining) |
|---|---|---|
| 0° | 100 Cycles, 85-2000F No Load | 91* 99* |
| 0° | 1000 Cycles, 85-2000F No Load | 95* 90* |
| 0° | 100 Cycles, 85-2200F No Load | 100* 95* |
| 0° | 1000 Cycles, 85-2200F No Load | 81* 78* |
| 0° | 1000 Cycles, 70-1200F 0-40 ksi | 96 |
| 0° | 1000 Cycles, 70-1200F 0-50 ksi | 93 |
| ±15° | 1000 Cycles, 70-1400F 0-40 ksi | 93 |

* From Reference 5.

## TABLE III

### Thermal Fatigue Data - (70-2000°F)

| Specimen Type | No. of Cycles |
|---|---|
| **Varying Fiber Diameter:** | |
| Large Outer Ply | 1000+ |
| Large Inner Ply | 1000+ |
| **Varying Volume Fraction*** | |
| Large Outer Ply | 830 |
| Large Inner Ply | 846 |
| $\pm 15°$ Angle Ply | 577 |

* Temperature Control Problems


Figure 13. Exploded View of Grip/Specimen Assembly After 1800F Test.

One approach commonly used to preclude such grip-area deformation is to water cool the grips. To ascertain the extent of the temperature gradient ($\Delta T$) this causes and the effect, if any, on properties, twin specimens contain-nominally 10v/o fiber were prepared and tested. Since FeCrAlY has a low shear strength, the grip ends of these specimens were of NiCrAlY, a higher shear strength matrix. The first was run at 1800F with no grip cooling. Thermocouples confirmed that both grips were within 10 degrees of the 1800F measured on the gage section.

In the run with cooled grips, the gage section was again determined to be 1800F by direct thermocouple reading; however, the grip temperature ranged from 1040 to 1170F.

Cross sectional areas of the two specimens were nearly identical, as were thicknesses. Fiber counts confirmed that the volume fractions were the same. However, measured tensile strengths were 72,300 and 61,400 psi for the cooled and uncooled specimens, respectively.

As is shown in Figure 14, failure occurred in the gage section in the cooled specimen and at the shoulder in the uncooled specimen.

This experiment shows that techique can and does influence test results.

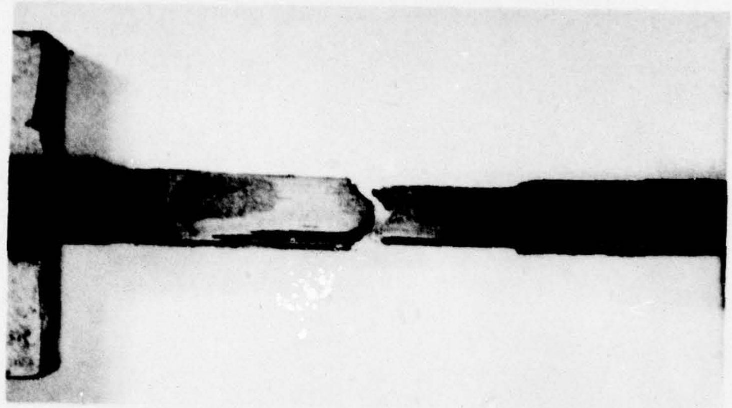
## 4.9 Advanced Reinforcement Material

An initial evaluation of hafnium carbide-coated SiC in FeCrAlY was performed. The HfC appears to bond readily to the FeCrAlY and is stable, at least through a typical processing cycle. However, as is shown in Figure 15, the fiber and coating are separated by a distinct void or very porous layer. This is not an isolated occurrence; every fiber exhibits this behavior.

A similar situation was encountered with tungsten wire coated with tantalum and hafnium carbide. As can be seen in Figure 16, a crack or disbond appears between the tantalum and the HfC layers. Again, the HfC is intact and appears to be well bonded to the FeCrAlY.

Resolution of this cracking problem was beyond the scope of this effort; however, potential for a HfC coating has been demonstrated.

Stress rupture tests were run on 27v/o tungsten-coated 5.6 mil SiC in FeCrAlY. Results are summarized in Table IV. Specimen AR-12 exhibited localized melting (approximately 14 fibers consumed) at one corner, as is shown in Figure 17. The Larson-Miller parameter of 51.7 is well below the extrapolated value of 59 for 30v/o W-1$ThO_2$/FeCrAlY but is about the same as that for DS MAR M-200



A. Grip and Gage Section at 1800F, Failed at Shoulder

B. Grip at ∿1100F, Gage at 1800F; Failed in Gage Section (Second Failure During Disassembly)

Figure 14. Tensile Failures from 1800F Tests.

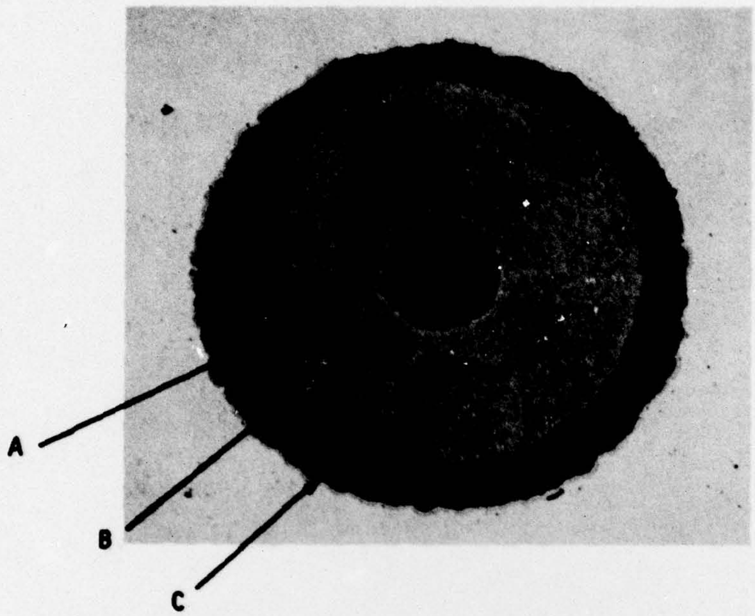



Figure 15. Hafnium Carbide-Coated SiC in FeCrAlY 400X

A - SiC Fiber
B - Interaction Zone
C - HfC

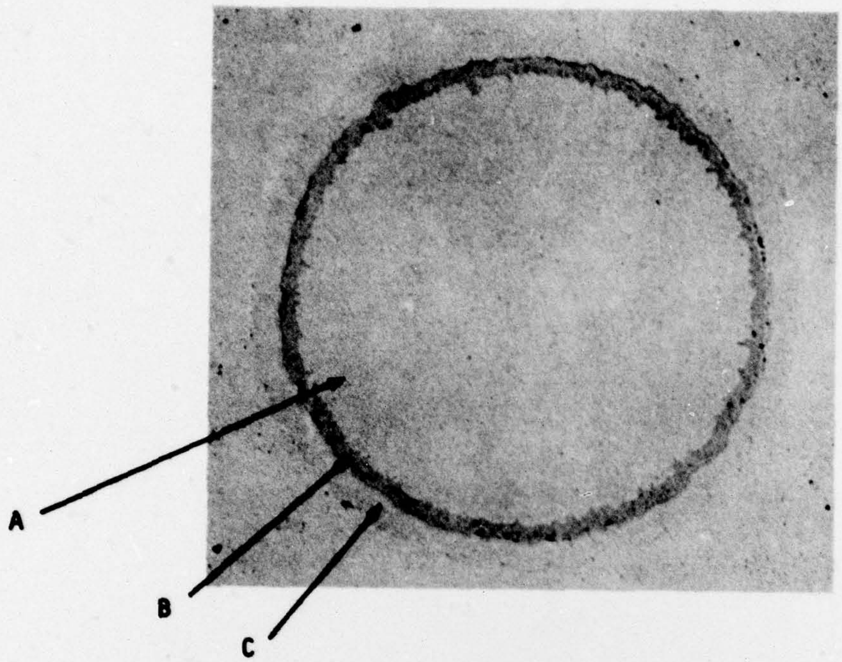



**Figure 16.** Hafnium Carbide (Over Tantalum Carbide)-Coated W in FeCrAlY. 400X

A - W Fiber
B - TaC
C - HfC

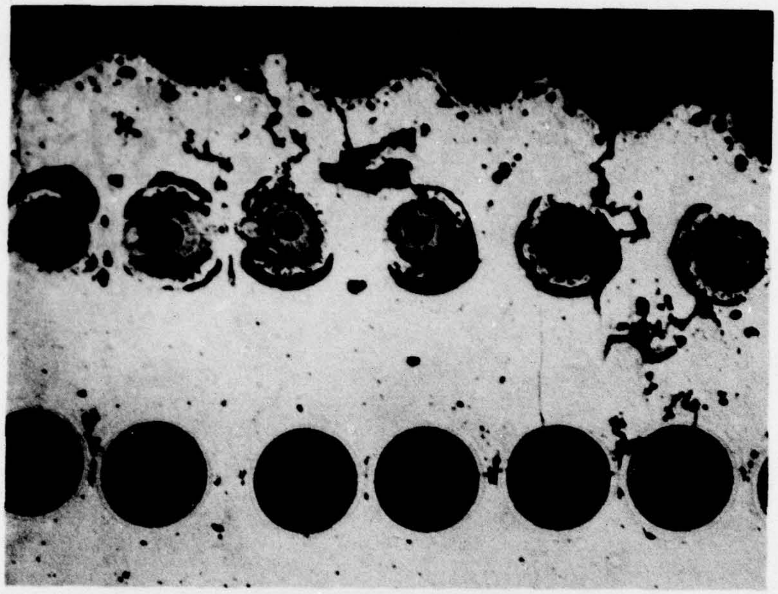


Figure 17. Specimen AR-13-1.

## TABLE IV

### Stress Rupture Data For Tungsten-Coated SiC/FeCrAlY

| | Stress (KSI) | | Temperature | Rupture Life | |
|---|---|---|---|---|---|
| Specimen | Section | Fiber | (°F) | Hr | P* |
| AR-12 | 20 | 75 | 2000 | 10.6 | 51.7 |
| AR-13-1 | 16 | 59 | 2000 | 11.8 | 51.8 |
| AR-13-2 | 15 | 56 | 1897 for 67.8 Hrs. then 2000 | 3.93 | 51.4 |

*P ≡ Larson-Miller Parameter, $T(20 + \log t_2) \times 10^{-3}$.

Specimen AR-13-1 failed in the gage section but exhibited delamination through internal ply 6 of the 8 plies constituting this panel. As is shown in Figure 17, the row of fibers immediately adjacent to the delamination was destroyed by reaction. It is unclear whether this represents a pre-existing defect or a consequence of the delamination during test, although the latter is more probable.

Specimen AR-13-2 was inadvertently run for 67.8 hours before an erroneous thermocouple junction was corrected and the temperature increased from 1897°F to the correct 2000°F. It then ran an additional 3.83 hours before failure.

The tungsten coating does protect the fibers, even in the vicinity of the overheated area (see Figure 18). Moreover, the stress cracks in the fibers generally do not penetrate through the coatings. While far from being an optimized system, this does show potential for coated SiC in FRS composites.

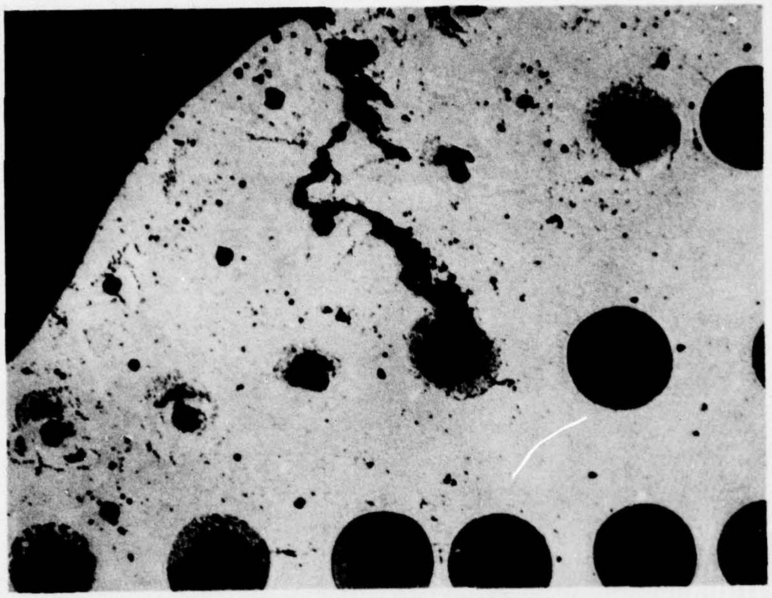


Figure 18. Specimen AR-12.

## 5.0 SUMMARY AND CONCLUSIONS

The objectives of this program were to continue development and characterization of W-1ThO$_2$/FeCrAlY composites which are intended for application in advanced gas turbine engines. Specifically the effect of varying fiber volume or diameter plus a cursory evaluation of coated SiC filament in FeCrAlY were addressed, together with feasibility of an ODS matrix alloy. Major conclusions are as follows

1. Within the range studied (20 to 50v/o), varying volume fractions in a symmetrical layup yield equivalent mechanical properties.

2. Within the range studied (0.004 to 0.010 inch), varying fiber diameters in a symmetrical layup give equivalent mechanical properties.

3. A shallow angle-ply ($\pm 15°$) composite tested in a double-notch shear configuration tends to fail in a mixed-mode, i.e., tension plus shear, at loads substantially greater than the pure shear values for unidirectional composites.

4. Based upon limited data, at 2000°F, $\pm 15°$ composites exhibit more rapid creep than unidirectional composites.

5. FRS composites can be produced from wrought sheet matrix materials; however, a thin powder cushion layer between sheets is quite beneficial in eliminating fiber damage.

6. Barrier coatings of HfC can prevent interaction betweek SiC filament and FeCrAlY, but properties of the resultant composites are well below expected values. An interaction zone between the fibers and coating appears to be partly responsible for these values.

7. Under certain conditions, FRS exhibits an anomalous creep behavior. This is most probably associated with residual stresses, but should be studied further.

## 6.0 RECOMMENDATIONS

The following actions are recommended.

1. Further characterize high temperature behavior of angle-plied laminates.
2. Improve the fabrication procedures for an ODS matrix FRS in order to enhance shear strength.
3. Establish cost-effective methods for the manufacture of FRS monotapes.
4. Further investigate the unusual creep behavior of certain FRS materials.

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